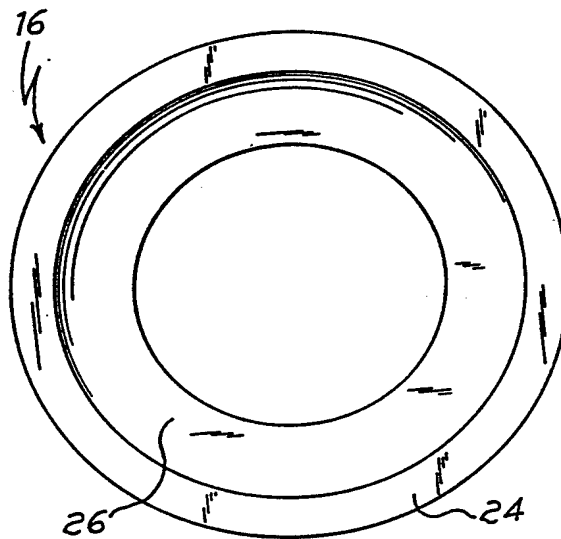


INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : H01J 7/18	A1	(11) International Publication Number: WO 94/02958 (43) International Publication Date: 3 February 1994 (03.02.94)
(21) International Application Number: PCT/IT93/00043 (22) International Filing Date: 3 May 1993 (03.05.93) (30) Priority data: MI92A001752 17 July 1992 (17.07.92) IT (71) Applicant: SAES GETTERS S.P.A. [IT/IT]; Via Gallarate, 215, I-20151 Milano (IT). (72) Inventors: FERRARIO, Bruno ; Via Melzi, 146, I-20027 Rescaldina (IT). MANINI, Paolo ; Via Mazzini, 104, I-20101 Arluno (IT). (74) Agents: ADORNO, Silvano et al.; Società Italiana Brevetti, Via Carducci, 8, I-20123 Milano (IT).		(81) Designated States: CA, JP, KR, RU, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>

(54) Title: HIGH-CAPACITY GETTER PUMP**(57) Abstract**

An improved high-capacity getter pump, comprising a plurality of porous sintered piled-up annuli made from a non-evaporable getter material and having: (i) a first planar surface having a central hole; (ii) a second planar surface, having a broader central hole, parallel to said first surface and spaced therefrom by a distance "d" of 1-10.5 mm; (iii) a third intermediate planar surface, interposed between said first and second surfaces, spaced from said first surface by a thickness "t" of 0.5-5.0 mm and having a hole coincident with the hole of said first surface; wherein the first surface of a subsequent annulus is in contact with the second surface of a preceding annulus, and wherein the first surface of a subsequent annulus is spaced from the third surface of a preceding annulus by a gas conductance having a height "c" of 0.5-10 mm.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	MR	Mauritania
AU	Australia	GA	Gabon	MW	Malawi
BB	Barbados	GB	United Kingdom	NE	Niger
BE	Belgium	GN	Guinea	NL	Netherlands
BF	Burkina Faso	GR	Greece	NO	Norway
BG	Bulgaria	HU	Hungary	NZ	New Zealand
BJ	Benin	IE	Ireland	PL	Poland
BR	Brazil	IT	Italy	PT	Portugal
BY	Belarus	JP	Japan	RO	Romania
CA	Canada	KP	Democratic People's Republic of Korea	RU	Russian Federation
CF	Central African Republic	KR	Republic of Korea	SD	Sudan
CG	Congo	KZ	Kazakhstan	SE	Sweden
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovak Republic
CM	Cameroon	LU	Luxembourg	SN	Senegal
CN	China	LV	Latvia	TD	Chad
CS	Czechoslovakia	MC	Monaco	TG	Togo
CZ	Czech Republic	MG	Madagascar	UA	Ukraine
DE	Germany	ML	Mali	US	United States of America
DK	Denmark	MN	Mongolia	UZ	Uzbekistan
ES	Spain			VN	Viet Nam
FI	Finland				

HIGH-CAPACITY GETTER PUMP

The present invention relates to an improved high-capacity getter pump, suitable for creating and maintaining the vacuum, for instance in an ultra-high vacuum chamber or in a high-energy particle accelerator.

Getter pumps are well known in the art and are suitable for creating and maintaining vacuum. The first commercially successful getter pump, described in US patent 3,780,501, was employing, in a housing, a pleated metal strip having a getter metal embedded therein. Additional examples of such getter pumps were described in US patents 3,609,064; 3,662,522; 3,961,897 and 4,137,012. Although these former getter pumps enjoyed a wide commercial success and market acceptance, they were still suffering from a drawback, residing in a limited sorption capacity inside a given volume.

In order to increase said sorption capacity, it was suggested to simply fill the pump housing with a getter material in the form of compressed pellets, having size and shape similar to the tablets used in the field of drugs; such pellets were typically showing a cylindrical shape, with a diameter of 5-10 mm and a height of 2-10 mm. However, when the housing is filled with such pellets, the access of the gas to the bulky getter

structure is far from being satisfactory. Another drawback, bound to the use of said pellets, was their tendency to produce undesired loose particles; moreover the bulky structure can show safety problems because of the possibility of a high exothermicity of the getter material, during possible ignitions, and this is true in particular when the used getter material has a low activation temperature.

Accordingly, it is a first object of the present invention to provide an improved getter pump substantially free from one or more of the drawbacks hereinabove.

Another object of the invention is to provide an improved getter pump having a higher sorption rate per unit volume, with respect to the getter pumps of the prior art.

A further object of the invention is to provide an improved getter pump having a higher sorption capacity per unit volume, with respect to the getter pumps of the prior art.

An additional object of the invention is to provide an improved getter pump resorting neither to pleated coated strips nor to pellets of getter material.

Other objects of the invention will be apparent to those of ordinary skill in the art, by reference to the following disclosure and drawings.

In its broadest aspect, the invention relates to an improved high-capacity getter pump, suitable for creating and maintaining the vacuum, for instance in a high-energy particle accelerator and in an ultra-high vacuum chamber, said pump comprising a plurality of porous sintered piled up annuli (flat disks) made from a non-evaporable getter material and having:

- i) a first planar surface having a central hole;
- ii) a second planar surface (having a broader central hole, with respect to said first surface) essentially parallel to said first surface and spaced therefrom by a distance "d" of about 1-10.5 mm (preferably 2-10 mm);
- iii) a third intermediate planar surface, essentially parallel to said first and second surfaces, interposed between said first and second surfaces, spaced from said first surface by a thickness "t" of essentially 0.5-5.0 mm and having a hole essentially coincident with the hole of said first surface;

wherein the first surface of a subsequent annulus is in contact with the second surface of a preceding annulus; wherein the first surface of a subsequent annulus is spaced from the third (intermediate) surface of a preceding annulus by a gas conductance (empty intermediate space), having a height "c" of 0.5-10 mm (preferably 1-5 mm) and wherein the values of "t", "d" and "c" are interrelated by the following equation:

- 4 -

$$d = t + c$$

Said gas conductances allow the gas molecules to enter the porous getter structure at a fast rate and the higher porosity of the porous sintered annuli better promotes the efficiency of the gas sorption (with respect to the pleated strips and to the pellets or tablets of the prior art).

Said annuli are suitably piled up in a housing, defining an inner channel with the edge of their holes. The getter pump according to the invention is furthermore equipped with a heater, for heating the annuli at the activation temperature and also at the desired operative temperature, and with a flange fastening said housing to a vacuum.

The porous sintered annuli of the pump according to the invention may have a shape selected from circular, elliptical, polygonal and combinations thereof (optionally tapered and/or bevelled). Moreover said annuli have a density from 1 to 5 g/cm³ and preferably from 1.5 to 3.5 g/cm³ and a surface area from 0.05 to 1 m²/g (preferably 0.1 - 1 m²/g).

The getter pump according to the present invention may be employed for maintaining the vacuum in a wide range of vacuum devices and apparatuses, for instance closed vacuum vessels (like e.g. a dewar or a vacuum jacket for a fluid transfer piping), particle accelerators (like for instance a synchrotron) and ultra-

high vacuum chambers. The new getter pumps can maintain a vacuum level as high as 10^{-6} and even 10^{-12} mbar (10^{-10} Pa).

5 A wide range of non-evaporable getter metals may be employed for the manufacture of the pumps according to the invention, for instance zirconium, titanium, hafnium, tantalum, thorium, uranium, niobium, mixtures thereof and alloys of these metals with each other and with other metals, such alloys being or being not intermetallic
10 compounds. These getter metals may be used alone or in admixture with other materials, like for instance antisintering agents. An exemplifying but not limiting series of non-evaporable getter metals for the manufacture of said porous sintered annuli comprises:

15 a) an alloy containing 84% Zr, balance Al, as described e.g. in US patent 3,203,901;

b) a metal composition according to US patent 3,584,253, based on Zr, Ta, Hf, Nb, Ti or U.

c) a metal composition according to example 3 of US
20 patent 3,926,832, based on a combination of Zr with a Zr-Al alloy;

d) the intermetallic compound Zr_2Ni described e.g. in US patent 4,071,335;

e) the Zr-M1-M2 alloys according to US patent
25 4,269,624, where M1 is V or Nb and M2 is Fe or Ni;

f) the Zr-Fe alloys according to US patent 4,306,887;

g) certain alloys of zirconium, vanadium and iron, as described in US patent 4,312,669, as well as other alloys of zirconium and vanadium and minor amounts of transition metals such as manganese;

5 h) certain alloys of zirconium, titanium and iron, as described in US patent 4,907,948.

According to a preferred embodiment of the present invention, said non-evaporable getter metal is selected from the Zr-V-Fe alloys and the Zr-Ti-Fe alloys, optionally in combination with Zr alone and/or Ti alone, these last being optionally in the form of hydrides. The combinations disclosed in GB Patent Application 2,077,487, in the name of the Applicant have proved to be particularly advantageous, being obtained from:

15 I) a ternary particulate Zr-V-Fe non-evaporable getter alloy having a composition (by weight) lying, when plotted on a ternary diagram, within a polygon having as its corners the following points (% b.w.):

- a) 75% Zr - 20% V - 5% Fe
- 20 b) 45% Zr - 20% V - 35% Fe
- c) 45% Zr - 50% V - 5% Fe

II) a particulate non-evaporable getter metal, selected from Zr and Ti, wherein the Zr and/or Ti particles have a smaller average size than the alloy particles.

25

Such combinations are traded by the Applicant as

"SAES St 172".

One advantageous method for manufacturing the porous sintered annuli of the pump according to the invention, starting from the combinations hereinabove, comprises the following steps:

- 5
- A) said non-evaporable getter metal is prepared in the form of a loose powder of Zr-V-Fe and/or Zr-Ti-Fe alloy particles, optionally in admixture with particles of Zr alone and/or Ti alone and with an expansion agent;
- 10
- B) said loose powder (or the consequent mixture) is poured in a mould and sintered at a temperature essentially comprised between 700 and 1200°C under an inert atmosphere (for instance argon).

15

Said sintering temperature of 700-1200°C, maintained for a time comprised between a few minutes and a few hours, is generally considered as a satisfactory one, whereas a lower temperature requires a longer time; the sintering time should give rise to a dimensional stability.

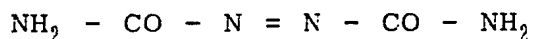
20

Said alloy particles have preferably a pre-sintering surface area equal to or higher than 0.15 and preferably 0.25 m²/g and a pre-sintering particle size up to 400 µm, preferably from 1 to 128 µm and even better from 1 to 50 µm. Said Zr and/or Ti particles, in their turn, have preferably an average particle size from 1 to 55 micrometer and a surface

25

area from 0.1 to 1.0 m²/g, wherein the weight ratio between the alloy particles and said Zr and/or Ti particles is suitably from 10 : 1 to 1 : 1.

The expansion agent may suitably be an inorganic and/or organic base containing nitrogen and/or phosphorus, which completely decomposes below the sintering temperature, for instance urea, azo-di-carbonamide and/or a carbamate like ammonium carbamate, in amounts from 0.1 to 15% b.w., with respect to the non-evaporable getter material (preferably 2 - 10%). The formula of azo-di-carbonamide is:



The heater may be arranged inside or outside the housing of the getter pump. The heating may be carried out by conduction or by radiation, for instance by means of a UHV quartz lamp.

The following drawings (Fig. 1-3) are supplied for illustrative purposes but do not limit in any way the scope of the invention; in particular:

Fig. 1 is a schematic representation of a getter pump according to the present invention in operating conditions;

Fig. 2 is an enlarged section view of a getter pump according to the present invention, taken along line II-II of Fig. 1;

Fig. 3 is a view of an annulus of a getter pump

- 9 -

according to the present invention.

Referring now to the drawings in general and in particular Figs. 1 and 2, there is shown an improved non-evaporable getter pump 10, having a gas-tight cylindrical housing 12 provided with a flange 14, which constitutes means for fastening said housing 12 to a vacuum vessel 15.

The getter pump 10 of Fig. 2 has a plurality of porous sintered annuli 16, 17, 18, 19, 20 piled up in said cylindrical housing 12, consisting of a non-evaporable getter metal. Each annulus has a first planar surface 22 and a second planar surface 24, essentially parallel to said first surface 22, spaced from the first surface by a distance "d" of about 1-10.5 mm.

Each annulus is furthermore showing an intermediate planar surface 26, essentially parallel to said first planar surface 22, interposed between first planar surface 22 and second planar surface 24.

Annuli 16, 17, 18, 19, 20 are piled up in the cylindrical housing 12, namely they are each other superimposed; the empty space (gas conductance) between the intermediate planar surface 26 of a preceding annulus and the first planar surface 28 of a subsequent annulus constitutes a gas conductance and the height of said conductance is from 0.5 to 10 mm (preferably 1-5 mm).

Getter pump 10 is equipped also with a thermocouple, not shown in the drawings, and with a coaxial inner

- 10 -

heater 30, which provides for the heating of annuli 17, 18, 19, 20, at the activation temperature (of the getter material) and also at the operative temperature.

5 The getter pumps according to the present invention have a sorption capacity several times greater, in a given volume, than the getter pumps of the prior art. Although the invention has been described in considerable detail with reference to certain preferred embodiments, it will be understood that many changes and modifications
10 can be carried out without departing from the scope of the invention.

CLAIMS

1. An improved high-capacity getter pump, suitable for creating and maintaining vacuum, comprising a plurality of porous sintered piled-up annuli made from a non-evaporable getter material and having:

- 5 i) a first planar surface having a central hole;
ii) a second planar surface (having a broader central hole, with respect to said first surface) essentially parallel to said first surface and spaced therefrom by a distance "d" of about 1-10.5
10 mm (preferably 2-10 mm);

- iii) a third intermediate planar surface, essentially parallel to said first and second surfaces, interposed between said first and second surfaces, spaced from said first surface by a thickness "t" of
15 essentially 0.5-5.0 mm and having a hole essentially coincident with the hole of said first surface;

wherein the first surface of a subsequent annulus is in contact with the second surface of a preceding annulus; wherein the first surface of a subsequent annulus is
20 spaced from the third (intermediate) surface of a preceding annulus by a gas conductance having a height "c" of 0.5-10 mm (preferably 1-5 mm) and wherein the values of "t", "d" and "c" are interrelated by the following equation:

25

$$d = t + c.$$

2. The pump of claim 1, wherein said annuli are

piled-up in a housing, defining an inner channel with the edge of their holes.

3. The pump of claim 1, equipped with a heater, for heating the annuli at the activation temperature and also at the desired operative temperature, and with a flange fastening said housing to a vacuum.

4. The pump of claim 1, wherein the porous sintered annuli have a shape selected from circular, elliptical, polygonal and combinations thereof (optionally tapered and/or bevelled) and have a density from 1 to 5 g/cm³ and preferably from 1.5 to 3.5 g/cm³ and a surface area from 0.05 to 1 m²/g (preferably 0.1 - 1 m²/g).

5. The pump of claim 4, wherein said non-evaporable getter material is selected from zirconium, titanium, hafnium, tantalum, thorium, uranium, niobium, mixtures thereof and alloys of these metals with each other and with other metals, such alloys being or being not intermetallic compounds, these metals being used alone or in admixture with other materials, like for instance antisintering agents.

6. The pump of claim 5, wherein said non-evaporable getter material is selected from the Zr-V-Fe alloys and the Zr-Ti-Fe alloys, optionally in combination with Zr alone and/or Ti alone, these last being optionally in the form of hydrides.

7. The pump of claim 6, wherein said non-evaporable

getter material is a combination of:

- 5 I) a ternary particulate Zr-V-Fe non-evaporable getter alloy having a composition (by weight) lying, when plotted on a ternary diagram, within a polygon having as its corners the following points (% b.w.):
- a) 75% Zr - 20% V - 5% Fe
 - b) 45% Zr - 20% V - 35% Fe
 - c) 45% Zr - 50% V - 5% Fe
- 10 II) a particulate non-evaporable getter metal, selected from Zr and Ti, wherein the Zr and/or Ti particles have a smaller average size than the alloy particles.

8. A method for manufacturing the porous sintered annuli of the pump of claim 6 comprising the following steps:

15

- A) said non-evaporable getter material is prepared in the form of a loose powder of Zr-V-Fe and/or Zr-Ti-Fe alloy particles, optionally in admixture with particles of Zr alone and/or Ti alone and with an expansion agent;
- 20
- B) said loose powder (or the consequent mixture) is poured in a mould and sintered at a temperature essentially comprised between 700 and 1200°C under an inert atmosphere (for instance argon).

25 9. The method of claim 8, wherein the alloy particles have a pre-sintering surface area equal to or higher than 0.15 and preferably 0.25 m²/g and a pre-

sintering particle size up to 400 μm , preferably from 1 to 128 μm and even better from 1 to 50 μm , wherein said Zr and/or Ti particles, in their turn, have an average particle size from 1 to 55 micrometer and a surface area from 0.1 to 1.0 m^2/g and wherein the weight ratio between the alloy particles and said Zr and/or Ti particles is suitably from 10 : 1 to 1 : 1.

10. The method of claim 8, wherein said expansion agent is an inorganic and/or organic base containing nitrogen and/or phosphorus, which completely decomposes below the sintering temperature, in particular urea, azo-di-carbonamide and/or a carbamate like ammonium carbamate, in amounts from 0.1 to 15% b.w., with respect to the non-evaporable getter material (preferably 2 - 10%).

Fig.1

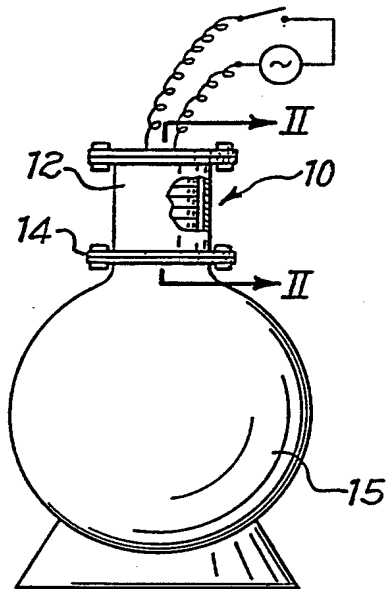


Fig.3

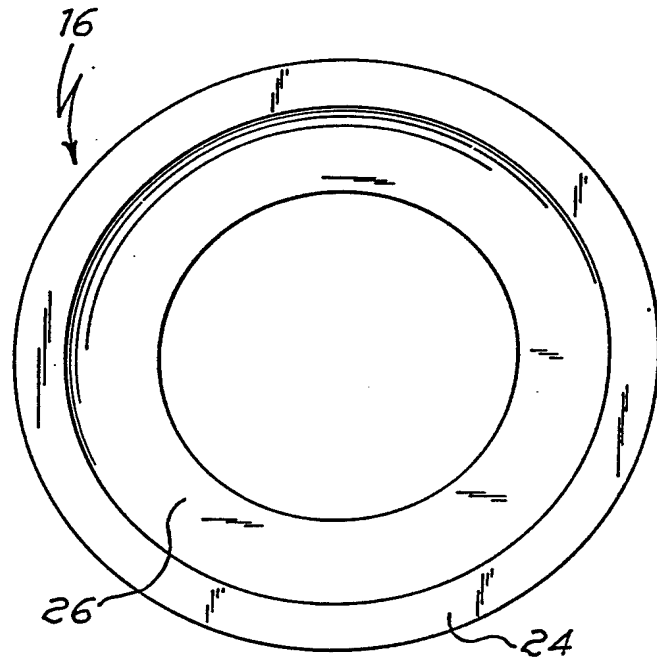
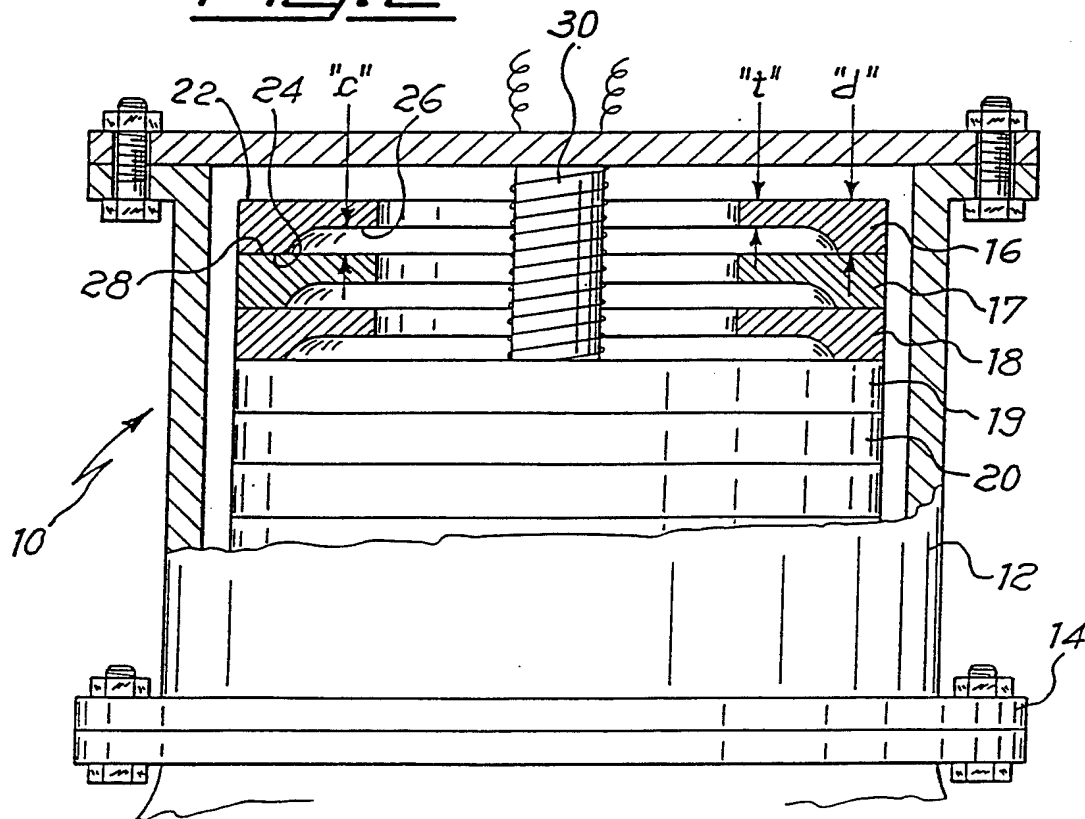


Fig.2



INTERNATIONAL SEARCH REPORT

PCT/IT 93/00043

International Application No

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)⁶

According to International Patent Classification (IPC) or to both National Classification and IPC
Int.Cl. 5 H01J7/18

II. FIELDS SEARCHED

Minimum Documentation Searched⁷

Classification System	Classification Symbols
Int.Cl. 5	H01J

Documentation Searched other than Minimum Documentation
to the Extent that such Documents are Included in the Fields Searched⁸

III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹

Category ^o	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
A	US,A,3 961 897 (T.A. GIORGI ET AL.) 8 June 1976 cited in the application see the whole document ---	1
A	US,A,3 305 290 (A. GANSSEN ET AL.) 21 February 1967 see column 3, line 71 - column 4, line 4 see figure 2 ---	1
A	GB,A,2 077 487 (SAES GETTERS) 16 December 1981 cited in the application see claims 1-10 ---	1,7,8
A	US,A,3 584 253 (M.WINTZER) 8 June 1971 cited in the application see the whole document -----	1,5

^o Special categories of cited documents : ¹⁰

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search 24 AUGUST 1993	Date of Mailing of this International Search Report 25 AUG 1993
International Searching Authority EUROPEAN PATENT OFFICE	Signature of Authorized Officer DAMAN M.A.

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

IT 9300043
SA 73796

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

24/08/93

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-3961897	08-06-76	DE-A, C 2446833	10-04-75
		FR-A, B 2246752	02-05-75
		GB-A- 1479613	13-07-77
		JP-C- 1137759	28-02-83
		JP-A- 50060813	26-05-75
		JP-B- 57030989	01-07-82
		NL-A- 7412719	03-04-75

US-A-3305290		None	

GB-A-2077487	16-12-81	DE-A, C 3122188	04-03-82
		FR-A, B 2484138	11-12-81
		JP-C- 1364249	09-02-87
		JP-A- 57053235	30-03-82
		JP-B- 61033613	02-08-86
		NL-A- 8102697	04-01-82

US-A-3584253	08-06-71	DE-A, C 1764092	17-02-72
		FR-A- 2005282	12-12-69
		GB-A- 1198600	15-07-70
		NL-A- 6904501	03-10-69
